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Introduction

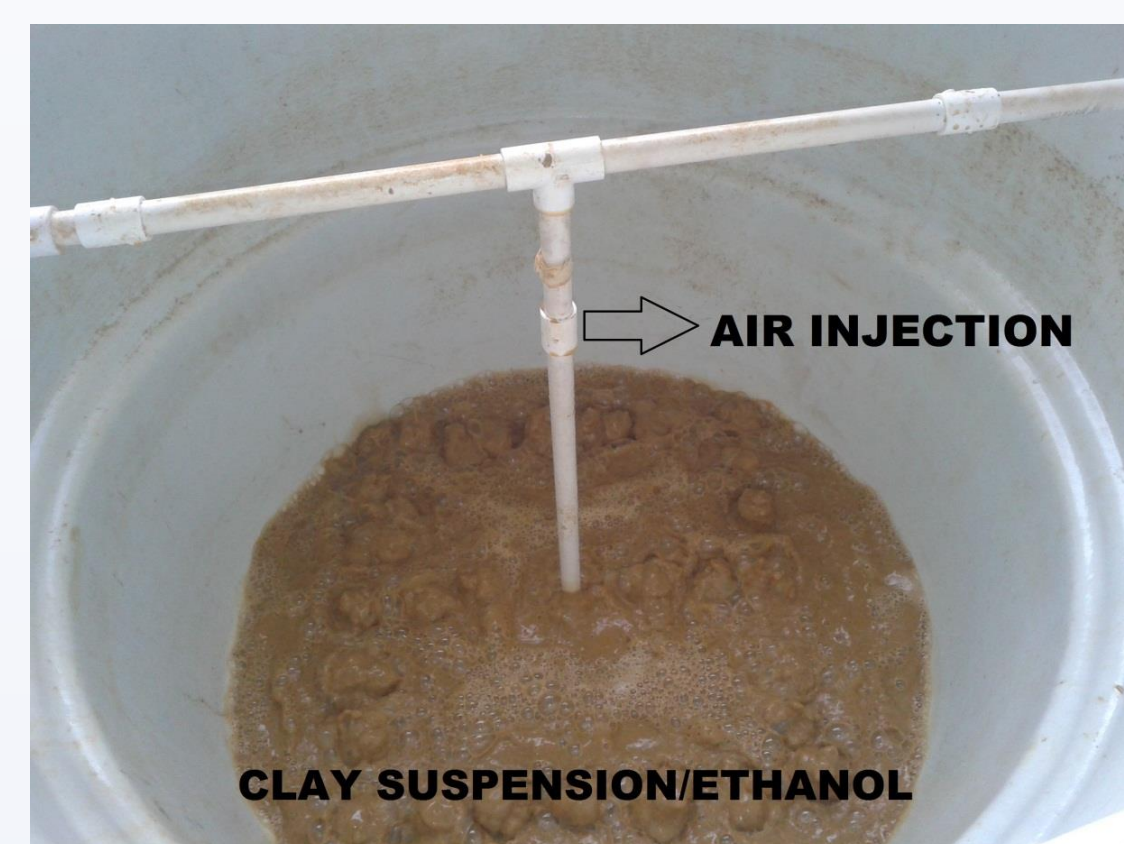
The preparation of Al/Fe-pillared clays (Al/Fe-PILC) has been extensively studied along the past few years because their low-cost, microporous structure and high catalytic performance in environmental applications e.g. for fixing by adsorption of different kinds of wastes, preparation of pharmaceuticals, catalytic removal of organic contaminants in aqueous effluents, formulation of coatings, among others^[1]. However, scaling-up of preparation and shaping (e.g. as extrudates, pellets, monoliths, etc) without significant loss of catalytic properties remains being rather a challenging issue^[2].

Experimental

Starting clay: Calcium-rich Bentonite (BVC) from Valle del Cauca - Colombia.

Synthesis of the Al/Fe-pillaring precursor (Al/Fe-Int)

Preparation was performed at 3.8 Kg scale by means of a procedure adapted from the already described elsewhere^[1]:



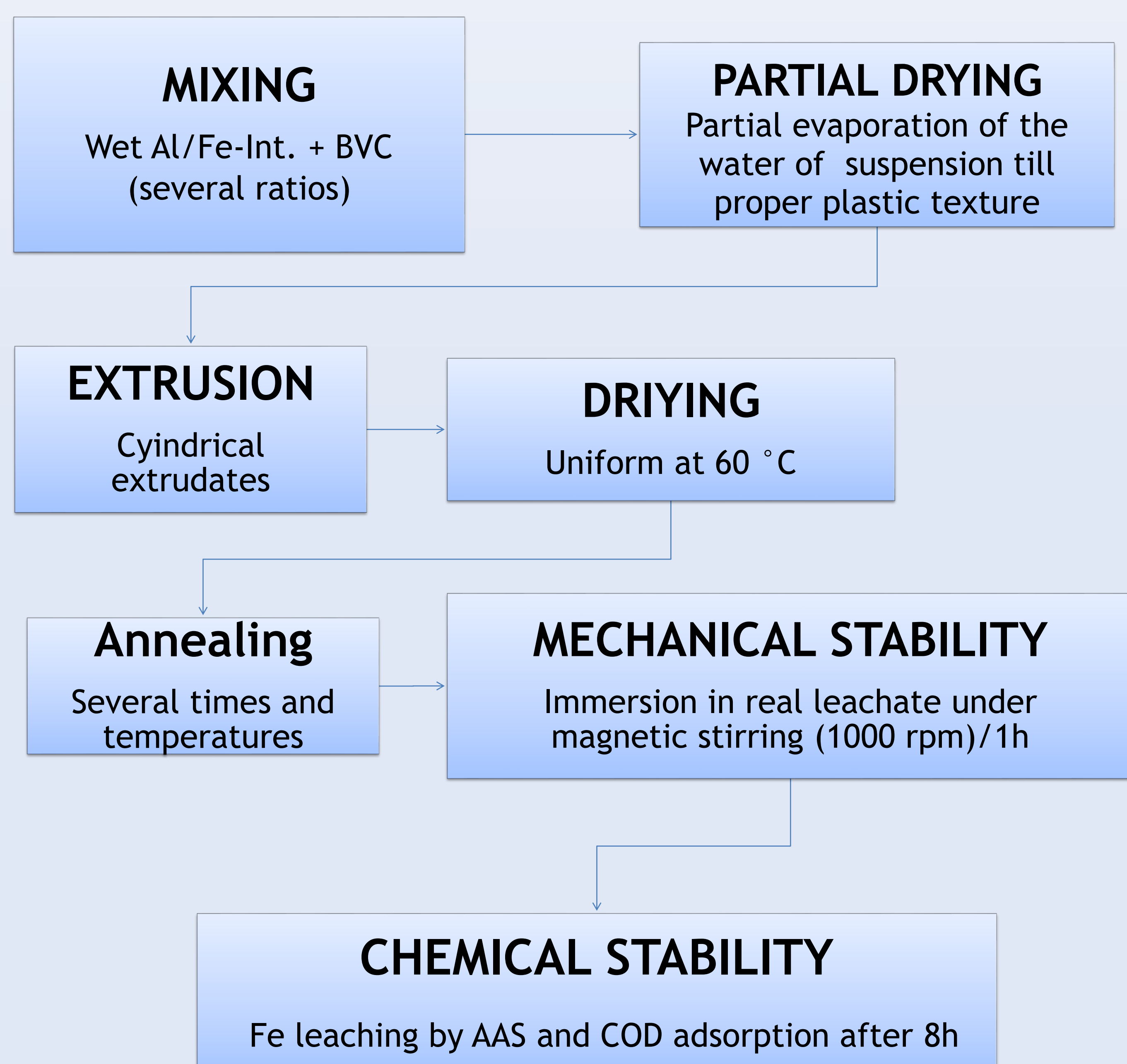
- Concentrated suspension: 50 % w/v of clay in ethanol (technical grade).
- Atomic Metal Ratio (Fe) (Fe/Al+Fe)*100 = 5.0 %
- Total Concentration of Metals [Al+Fe] = 0.6 mol/L
- Hydrolysis Ratio (OH⁻/Al+Fe) = 1.6

Figure.1 Preparation of the Al/Fe-pillaring precursor

Characterization

(i) Elemental analysis (XRF) ; (ii) Cationic exchange capacity (CEC) - (NH₄)⁺-exchange and (iii) X-ray diffraction

Preparation and analysis of extrudates



Results

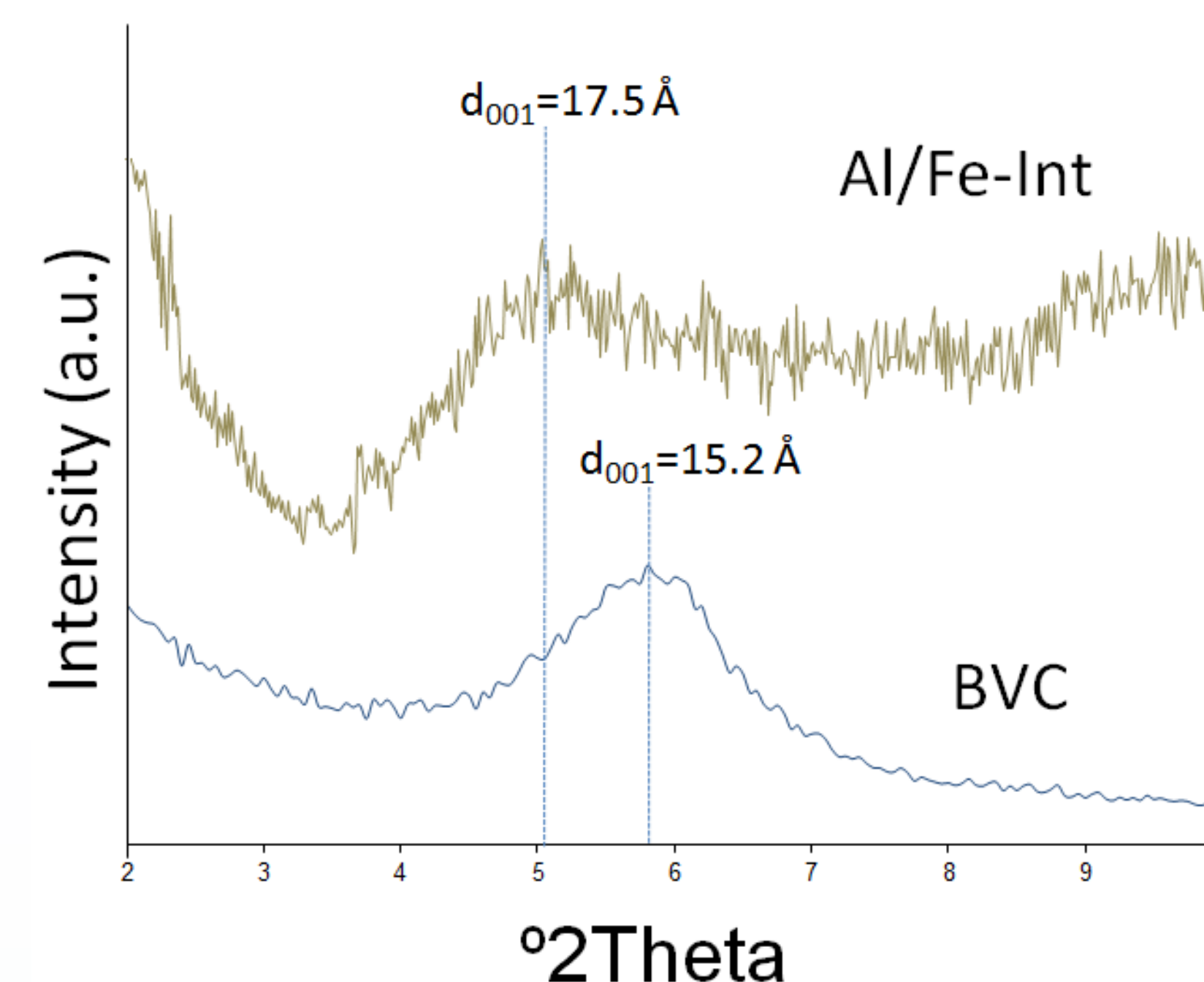


Figure.3 Powder small-angle XRD of raw bentonite (BVC) and interlayered bentonite (Al/Fe-Int).

Table 1. Main physicochemical properties of starting and Al/Fe - pillared clay^(a)

Sample	SiO ₂ (% w/w)	Al ₂ O ₃ (% w/w)	Fe ₂ O ₃ (% w/w)	CEC (meq./100g clay)
BVC	46.0	20.3	8.50	64
Al/Fe-BVC ^(b)	49.7	26.8	11.2	38

^(a) Dry basis (110 °C); ^(b) Calcined at 500 °C/2 h.

Table 2. Mechanical and chemical stability of extruded materials after testing

Composition ratio Al/Fe-Int:BVC:water +	Thermal Treatment	Mechanical Testing ^(a)	Chemical Stability	
	T/t (°C/h)	Particle sizes over sieve 16 (w/w %) ^(b)	[Fe] (ppm)	COD adsorbed (mg O ₂ /g extr.)
31:31:38	500/2	97	0.5	24
31:31:38	400/2	17	ND	-----
43:19:38	500/2	96	0,3	21
43:19:38	400/2	97	ND	41
50:12:38	500/2	94	1.0	17
50:12:38	400/2	-----	ND	-----
56:6:38	500/2	90	0.7	0
56:6:38	400/2	-----	ND	-----
59:3:38	500/2	84	ND	0
59:3:38	500/4	82	ND	35
61:1:38	500/2	87	ND	11
61:1:38	500/4	85	ND	0
62:0:38	500/2	86	0.7	18
62:0:38	400/2	-----	1.0	-----

^(a) Immersion in leachate 1h/ 1000 rpm; ^(b) Sieve ASTM 16 = ($\phi > 1.18$ mm); ND = Not detected (AAS).

Conclusion

The pillaring was successful at 3.8 Kg scale (see Fig. 3 and Table 1). Heating at 500 °C produced the best mechanical properties in the extrudates irrespective the ratio Al/Fe-Int:BVC (active phase:binder); besides, these extrudates exhibited higher capacity of adsorption when the fraction of the binder (raw clay) was increased. A mass ratio 43/19/38 for active phase/binder/water was found to be the optimal in terms of both, mechanical and chemical stability (against Fe leaching) of the extrudates.



Figure 2 Final extrudates

Acknowledgement

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References

- [1] Galeano L.A., Vicente M.A., Gil A., *Catal. Rev. Sci & Eng.* 56 (2014) 239- 287.
- [2] Mohino F., Martin A.B., Salerno P., Bahamonde A., Mendioroz S. *Appl. Clay Sci.* 29(2) (2005) 125-136.